A Study of Kapton Degradation Under Simulated Shuttle Environment

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I. INTRODUCTION

Weight loss and severe degradation of the surface of Kapton and other materials occur in low earth orbit. Atomic oxygen, the major ambient species at low earth altitude and incident with approximately 5 eV energy in ram conditions, is the primary suspect, but a thorough study of oxygen-Kapton interactions has not yet been carried out.

Because of the unexpected severity of this degradation as well as the lack of fundamental understanding of the mechanism involved, it is important to determine the quantum yields for the individual fluences that make up the low earth orbit so they may be compared with the spacecraft data. In addition to approximating the individual components, one must also simulate the possible synergistic effects of UV, higher temperatures from the solar radiation and, of course, the atomic and molecular beams.

The kinetic energy with which particles strike the shuttle is well below the threshold for appreciable direct physical removal of material (sputtering). Thus, the basic interaction responsible for the observed degradation must be chemical. This has led some researches to conclude that the kinetic energy is irrelevant and that thermal atomic oxygen would produce comparable degradation. However, the data to date do not rule out a direct energy enhancement of the interaction, and, in addition, this kinetic energy could well increase the net rate of degradation by increasing the rate of removal of adsorbed surface layers of the gaseous products of the interaction (such as CO).

The present research employs a low-energy ion source to simulate the shuttle low earth orbit environment. This source, together with diagnostic tools including surface analysis and mass spectroscopic capability, is being used to carry out experiments from which quantum yields may be

obtained. In the following report we discuss developments in the work and its current status as well as plans for the future.

II. INSTRUMENTATION

The unique assembly of instrumentation available in our laboratory for this study includes: 1) an ultra-high-vacuum environment in which *in situ* surface analysis on insulating specimens can be carried out immediately following oxygen bombardment; 2) the capability of mass spectroscopic analysis of the particles leaving the sample surface during oxygen bombardment; and 3) a low-energy oxygen gun with a characterized beam and particle fluxes and energies comparable to those seen by the space shuttle in low earth orbit.

Our gun (developed by Kimball Physics, Inc., of Wilton, New Hampshire) produces a beam of oxygen ions with an energy variable from 500 eV to less than 5 eV. At this lowest energy the energy spread of the ions is approximately 3 eV, the maximum ion current is approximately 0.1 microamperes, and the beam diameter is about 2 millimeters. About 70% of the beam is O_2^+ and 30% O^+. Figure 1 shows typical traces of ion current to a biased collector as a function of retarding voltage.

III. RESEARCH DEVELOPMENTS AND CURRENT STATUS

In the initial stages of this research our plans were to bombard Kapton at various ion energies and observe the resultant surface degradation using standard "visual" techniques such as scanning electron microscopy. However, the maximum ion flux produced by our gun ($\sim 2 \times 10^{13}$ ions/cm^2-sec) is substantially less than what we had anticipated, based on our preliminary design discussions with Kimball Physics, and only about 1/30 of the oxygen atom flux experienced by the shuttle in low earth orbit.

With this reduced flux, producing visible surface damage would require long periods of bombardment ($\sim$ 200 hours), an especially unappealing prospect in light of our experience that our gun filament has a lifetime of only $\sim$ 100 hours at the O_2 pressure necessary to produce a 0.1 microampere beam. Therefore, we decided to pursue instead a technique based on mass spectrometer analysis of the gaseous products of oxygen ion bombardment.

The quadrupole mass spectrometer has been repositioned so that its electron bombardment ionizer is only $\sim$ 3 cm from the surface being bombarded by our oxygen ion beam. Figure 2 shows the present arrangement of the essential components within our vacuum envelope. With the mass spectrometer set to detect a particular gaseous product, the oxygen ion beam is square wave amplitude modulated by a voltage applied to the gun's focus electrode and the modulated component of the mass spectrometer signal is synchronously detected with a lockin detector. A test of the sensitivity of this technique has been carried out by monitoring the CO signal while bombarding a carbon target. A small, but reproducible, signal has been observed when bombarding the carbon and <u>also</u> when bombarding an empty space on the sample carousel or a copper plate normally used for profiling the oxygen ion beam. To determine whether these unexpected signals were due to oxygen interaction with surface carbon contamination or to oxygen ion desorption of CO adsorbed on these surfaces, we first established by taking Auger electron spectra that these surfaces were indeed contaminated with carbon. Then, we replaced the O_2 supply for the ion gun with a source of argon. With modulated argon ion bombardment we observed no modulated CO signal from the mass spectrometer when the target was carbon, an empty space on the carousel, or the copper profiling plate. We conclude

from this that all of the CO signals seen under oxygen ion bombardment are due to oxygen interaction with surface carbon and not to ion desorption of adsorbed CO molecules.

Figure 3 shows typical strip chart recorder traces of the lockin detector output as a function of time taken for different ion gun electron emission current settings (and, thus, different ion currents from the gun) and lockin sensitivity settings. Each trace starts with a period of approximately 1 minute during which the electron emission current is zero. This establishes a zero signal baseline from which the ion induced signal can be measured. The emission is then turned up (the ON portion of each trace) for approximately 1 minute and then reduced again to zero to check for any baseline drift. During the ON portion of each trace the carbon target is being bombarded by a 32 Hz square pulse ion current with a 50% duty cycle. For trace number 7 the 1 mA electron emission yields ion pulses of 0.13 μA. The on-off displacement in this trace corresponds to an increase in CO pressure at the mass spectrometer of 5×10^{-12} torr. The fact that this data was taken when the CO background pressure in our vacuum system was approximately 2×10^{-9} torr is a measure of the sensitivity of the modulation technique. The noise in the traces of Figure 3, which were taken with a lockin time constant of 1 second, is due entirely to the statistical fluctuations in the large CO background signal from the mass spectrometer.

We have established that at these low ion energies surface charging of Kapton is immediate and "permanent", i.e., Kapton retains its charge until neutralized by irradiation with electrons. This means that it is not possible to observe ion induced signals from Kapton without a

source of neutralizing electrons. Electron leakage from the electron bombardment region of our mass spectrometer, which is only 3 cm from our target, is quite sufficient for this charge neutralization, and we have seen CO signals from oxygen ion bombardment of Kapton which are comparable in strength to those produced with a carbon target. However, since these electrons have a broad distribution in energy (up to 70 eV), the surface potential of the Kapton (and therefore the kinetic energy with which the oxygen ions strike the surface) is not well defined.

We have submitted the following abstract to the 32nd National Symposium of the American Vacuum Society:

CARBON MONOXIDE PRODUCTION IN LOW ENERGY OXYGEN ION BOMBARDMENT OF PYROLYTIC GRAPHITE AND KAPTON SURFACES.* C.C.Horton, T.G.Eck, R.W.Hoffman, Dept. of Physics, Case Western Reserve University, Cleveland, Ohio, 44106. We report the interaction of low energy oxygen ions with pyrolytic graphite and Kapton surfaces using modulated ion beam mass spectrometry. The oxygen ions, predominantly O_2^+, were varied in energy from 5 to 100 eV with beam currents of 100 nA in a 2 mm diameter spot (flux $=2\times10^{13}$ ions/cm^2-sec). The energy spread at 5 eV was about 3 eV. The square wave modulated ion beam was incident normal to the surface and a mass spectrometer was tuned to CO. The CO signal was small compared to the background CO pressure and a lock-in amplifier was used to measure the amplitude. The CO was produced on the pyrolytic graphite surface with a quantum yield between 10^{-1} and 1 CO molecule per incident O_2^+, and was dependent on the initial oxygen coverage. Comparable yields were obtained for Kapton. The yields increased slightly as the ion energy was increased. The oxygen bombardment of Kapton stimulates the degradation of Kapton thermal blankets on the Space Shuttle in low earth orbit.

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IV. PLANS FOR THE FUTURE

During the period of our grant renewal we will

(1) complete our investigation of oxygen ion bombardment of carbon by observing the CO signal as a function of ion energy and beam modulation frequency,

(2) extend our measurements to Kapton by installing in our system a

source of low energy electrons and screen grids to prevent electrons from the mass spectrometer from reaching the target,

(3) investigate the effects of sample heating and UV irradiation, both separately and together, during low-energy ion bombardment,

(4) look for signals with argon ions from our gun together with O_2 backfill of our vacuum system.

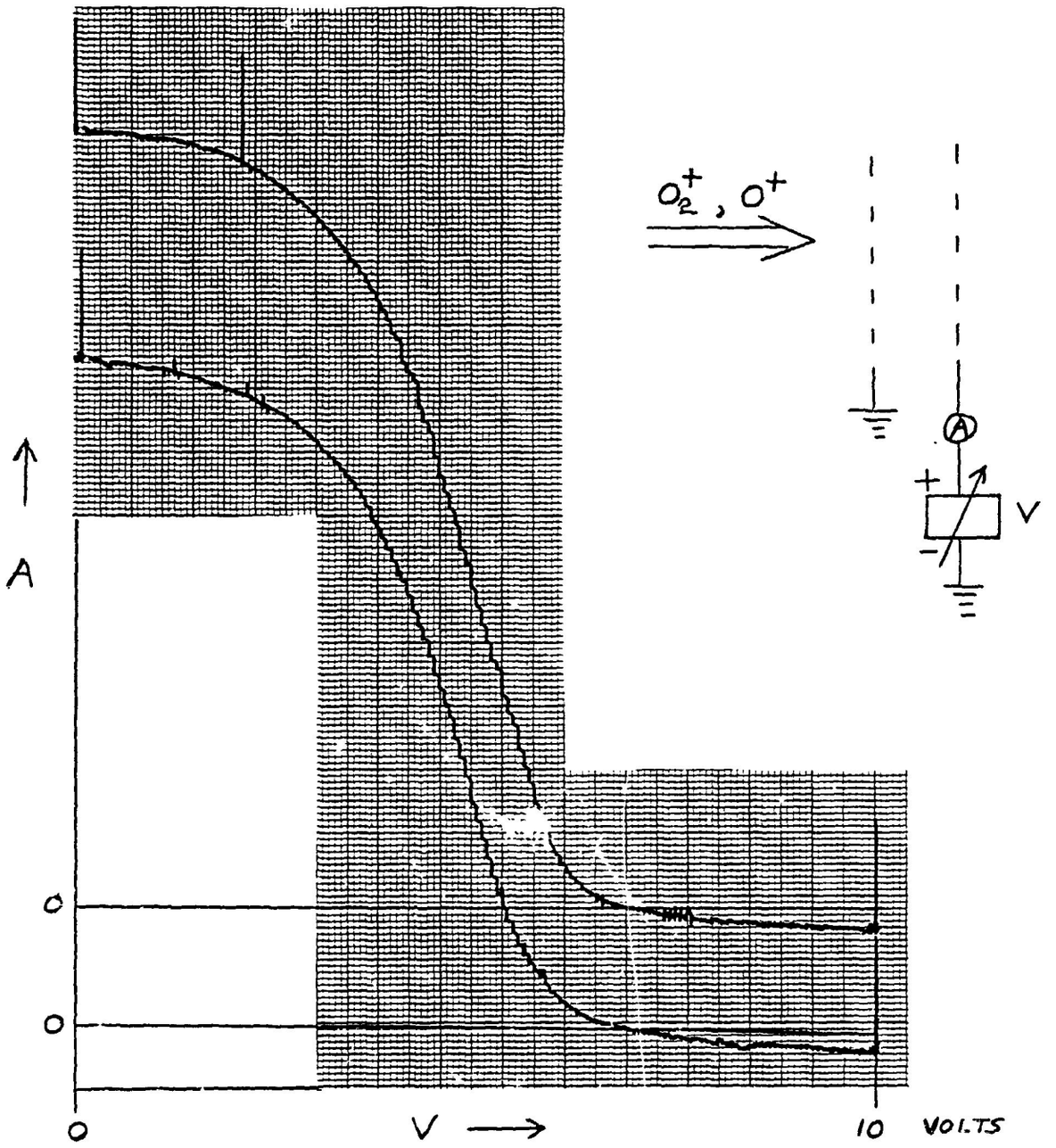

Figure 1

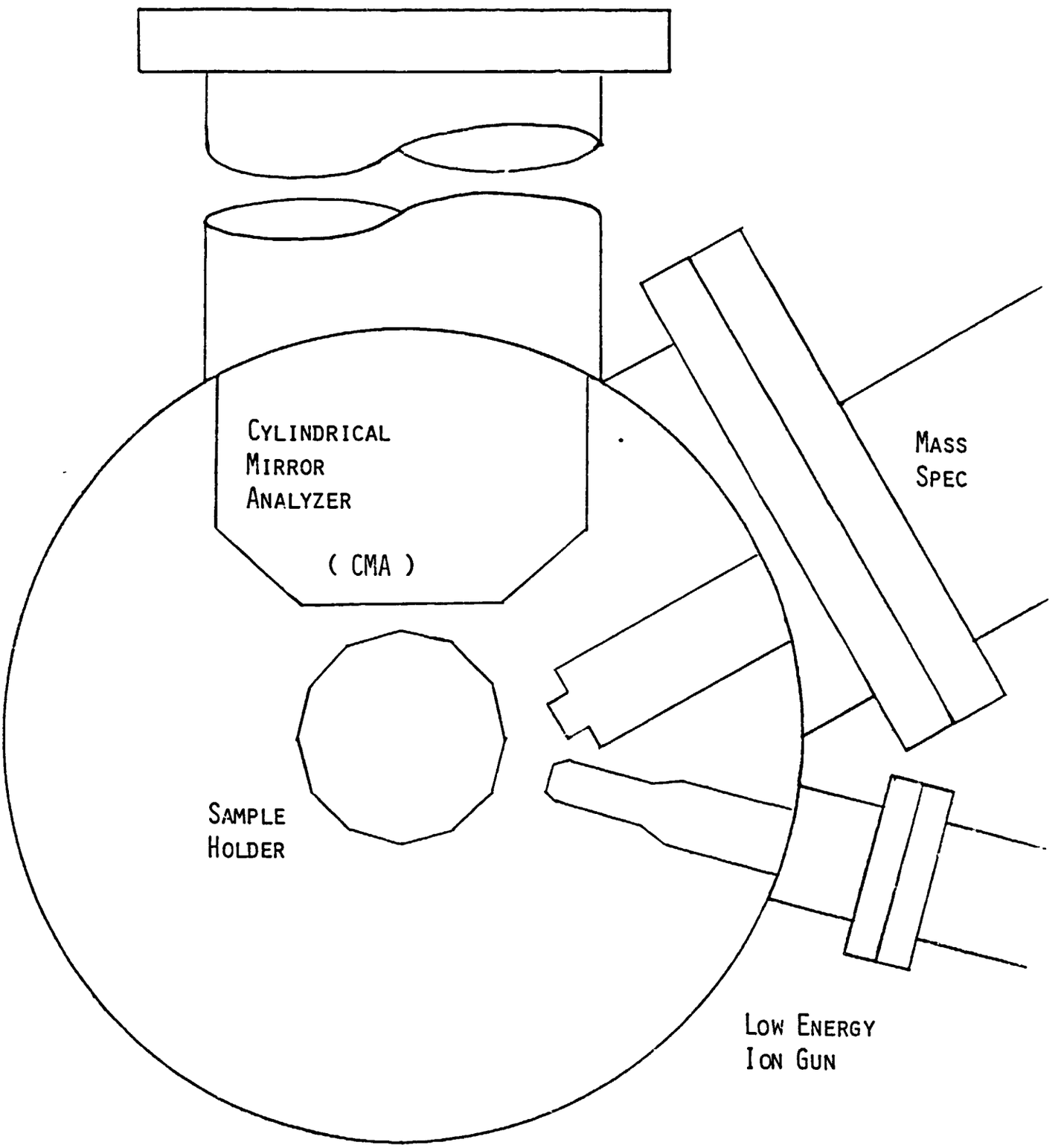
CYLINDRICAL
MIRROR
ANALYZER
(CMA)
MASS
SPEC
SAMPLE
HOLDER
LOW ENERGY
ION GUN

Figure 2

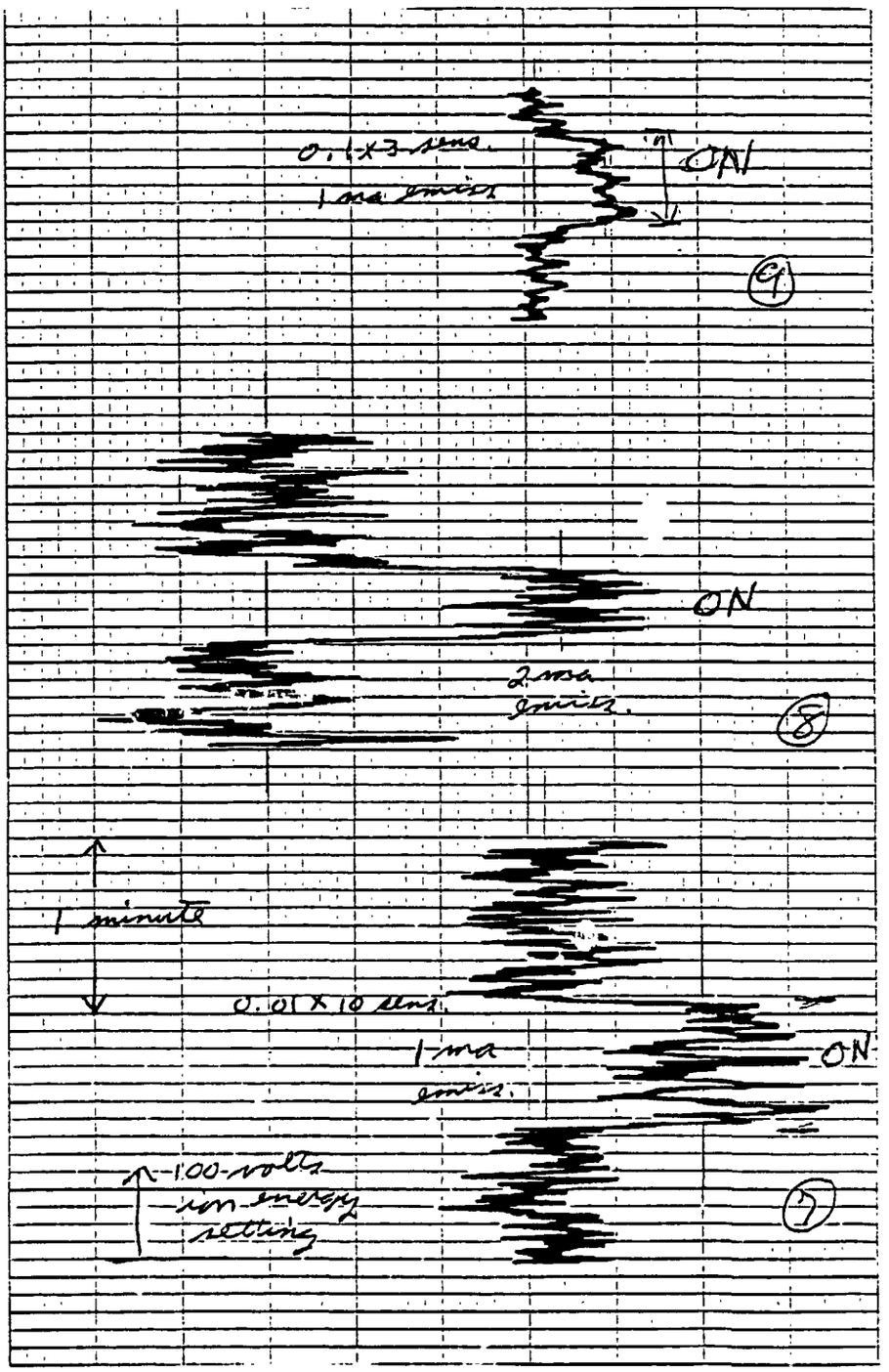

Figure 3